NATL INST. OF STAND & TECH
A11107 237357
NIST
PUBLICATIONS

REFERENCE

A UNITED STATES
DEPARTMENT OF
COMMERCE
PUBLICATION

National Bureau of Standards
Library, E-01 Admin. Bldg.
OCT 6 1981
191101
QC
100
.U5753

NBS TECHNICAL NOTE 613

Martensite Transformation Detection in Cryogenic Steels (Magnetometer Development)

U. S.
EPARTMENT
OF
COMMERCE
QC
100
.U6753
No.613
1971

NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards[1] was established by an act of Congress March 3, 1901. The Bureau's overall goal is to strengthen and advance the Nation's science and technology and facilitate their effective application for public benefit. To this end, the Bureau conducts research and provides: (1) a basis for the Nation's physical measurement system, (2) scientific and technological services for industry and government, (3) a technical basis for equity in trade, and (4) technical services to promote public safety. The Bureau consists of the Institute for Basic Standards, the Institute for Materials Research, the Institute for Applied Technology, the Center for Computer Sciences and Technology, and the Office for Information Programs.

THE INSTITUTE FOR BASIC STANDARDS provides the central basis within the United States of a complete and consistent system of physical measurement; coordinates that system with measurement systems of other nations; and furnishes essential services leading to accurate and uniform physical measurements throughout the Nation's scientific community, industry, and commerce. The Institute consists of a Center for Radiation Research, an Office of Measurement Services and the following divisions:

Applied Mathematics—Electricity—Heat—Mechanics—Optical Physics—Linac Radiation[2]—Nuclear Radiation[2]—Applied Radiation[2]—Quantum Electronics[3]—Electromagnetics[3]—Time and Frequency[3]—Laboratory Astrophysics[3]—Cryogenics[3].

THE INSTITUTE FOR MATERIALS RESEARCH conducts materials research leading to improved methods of measurement, standards, and data on the properties of well-characterized materials needed by industry, commerce, educational institutions, and Government; provides advisory and research services to other Government agencies; and develops, produces, and distributes standard reference materials. The Institute consists of the Office of Standard Reference Materials and the following divisions:

Analytical Chemistry—Polymers—Metallurgy—Inorganic Materials—Reactor Radiation—Physical Chemistry.

THE INSTITUTE FOR APPLIED TECHNOLOGY provides technical services to promote the use of available technology and to facilitate technological innovation in industry and Government; cooperates with public and private organizations leading to the development of technological standards (including mandatory safety standards), codes and methods of test; and provides technical advice and services to Government agencies upon request. The Institute also monitors NBS engineering standards activities and provides liaison between NBS and national and international engineering standards bodies. The Institute consists of the following technical divisions and offices:

Engineering Standards Services—Weights and Measures—Flammable Fabrics—Invention and Innovation—Vehicle Systems Research—Product Evaluation Technology—Building Research—Electronic Technology—Technical Analysis—Measurement Engineering.

THE CENTER FOR COMPUTER SCIENCES AND TECHNOLOGY conducts research and provides technical services designed to aid Government agencies in improving cost effectiveness in the conduct of their programs through the selection, acquisition, and effective utilization of automatic data processing equipment; and serves as the principal focus within the executive branch for the development of Federal standards for automatic data processing equipment, techniques, and computer languages. The Center consists of the following offices and divisions:

Information Processing Standards—Computer Information—Computer Services—Systems Development—Information Processing Technology.

THE OFFICE FOR INFORMATION PROGRAMS promotes optimum dissemination and accessibility of scientific information generated within NBS and other agencies of the Federal Government; promotes the development of the National Standard Reference Data System and a system of information analysis centers dealing with the broader aspects of the National Measurement System; provides appropriate services to ensure that the NBS staff has optimum accessibility to the scientific information of the world, and directs the public information activities of the Bureau. The Office consists of the following organizational units:

Office of Standard Reference Data—Office of Technical Information and Publications—Library—Office of Public Information—Office of International Relations.

---

[1] Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D.C. 20234.
[2] Part of the Center for Radiation Research.
[3] Located at Boulder, Colorado 80302.

NAL BUREAU OF STANDARDS
DEC 2 8 1971
not acc
QC100
U5753
No. 613
1971

UNITED STATES DEPARTMENT OF COMMERCE
Maurice H. Stans, Secretary
U.S. NATIONAL BUREAU OF STANDARDS ● Lewis M. Branscomb, Director

NBS TECHNICAL NOTE 613

ISSUED DECEMBER 1971

Nat. Bur. Stand. (U.S.), Tech. Note 613, 31 pages (Dec. 1971)
CODEN: NBTNA

Martensite Transformation Detection in Cryogenic Steels (Magnetometer Development)

F. R. Fickett

Cryogenics Division
Institute for Basic Standards
National Bureau of Standards
Boulder, Colorado 80302

Sponsored by
Naval Air Engineering Center
Philadelphia, Pennsylvania
Under Project Order 1-8022

NBS Technicol Notes ore designed to supplement the Bureou's regular publicotions progrom. They provide o meons for moking ovoiloble scientific doto that ore of tronsient or limited interest. Technicol Notes moy be listed or referred to in the open literoture.

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402
(Order by SD Catalog No. C13.46:613), Price 40 cents

Contents

		Page
1.	Introduction	1
2.	Constraints	3
3.	Systems Studied	4
4.	Theory of Operation	6
5.	Design Criteria	8
	5.1 The Three Components of the Magnetometer Head	9
	5.2 Temperature Effects	12
	5.3 Shock Effects.	13
	5.4 Electrical	13
	5.5 Electronics	13
6.	Operating Characteristics	16
7.	Further Development	23
8.	References	25
Appendix		26

List of Figures

Figure 1.	Magnetometer head and magnetization sequence.	7
Figure 2.	Circuit diagram for resistive voltage offset zero.	15
Figure 3.	Sensitivity of bismuth probe (typical).	19
Figure 4.	Detection of percent martensite by bismuth probe (typical).	20
Figure 5.	Sensitivity of semiconductor probe (typical).	21
Figure 6.	Detection of percent martensite by semiconductor probe (typical).	22
Figure 7.	In-place probe calibration technique.	27

Martensite Transformation Detection in Cryogenic Steels

(Magnetometer Development)

F. R. Fickett

This report presents design criteria for a magnetometer device developed to determine the martensite concentration in 304 stainless steel. Specifically, the device is designed to monitor the cold tank of an airborne liquid oxygen container which is subject to periodic stress pulses. The amount of martensite is relatable to the plastic strain and thus provides a mechanism for the prediction of impending failure

The magnetometer, developed after a critical review of the constraints, is an excitation coil-Hall effect system. The system is very versatile in design and is easily adapted to particular problems. It is relatively insensitive to thermal and mechanical shock and easily capable of detecting martensite at the 1% level.

Key words: Fatigue monitor; low temperature; magnetometer; martensite; nondestructive.

1. Introduction

For a variety of reasons AISI 304 stainless steel is used as the primary construction material in large, airborne liquid oxygen tanks. These tanks are subject to repeated severe shocks, both while warm and while at liquid oxygen temperature (90 K). The repeated shocks may lead to fatigue fracture in regions of high stress concentration such as the vicinity of the (relatively few) support rods for the inner tank.

Under stress, and particularly at low temperatures, 304 stainless steel, which has an austenitic structure, transforms partially to a bcc martensitic phase, α', which, unlike austenite, is ferromagnetic. A

previous project [1][1] by our group has investigated the relationship of martensite formation to plastic strain under shock loading at various temperatures down to 76 K and Reed and co-workers [2-5] have investigated the formation of the martensitic phase in detail. One should stress their result: there is no indication that formation of the martensitic phase itself leads to failure of the steel; in fact, it may well strengthen the steel at temperatures T > 76 K.[2] The amount of martensite present following repeated stress pulses does, however, provide a measure of the amount of plastic strain and thus is a monitor of the fatigue process. The martensite can be detected by specialized magnetometry.

The purpose of the project, for which this paper is the final report, was to investigate various methods of monitoring the relative amount of martensite present at high stress points on liquid oxygen tanks and to arrive at a design concept for a magnetometer system.

Magnetometers of many different designs exist in profusion and, in fact, one of the early parts of this project involved a survey of existing devices in both commercial and research laboratory use to determine if any of these would be suitable for this application.

In the next section we discuss the constraints on the device unique to this application. Section 3 reviews the approaches studied. Section 4 presents the theory of the detector and section 5 gives some design criteria. Section 6 gives the operating characteristics of a laboratory model and section 7 discusses the steps necessary in order to produce a working instrument from the design criteria.

---

[1] Figures in brackets indicate the literature references at the end of this paper.

[2] Similarly one should be aware that, at low temperatures, say near that of liquid hydrogen (20 K), the martensite formation may weaken the steel [5, 6].

2. Constraints

Two major classes of magnetometers were considered: 1) large area detectors which could scan the entire inner tank and would not be physically attached and 2) small area detectors which would be attached to the tank at points of high stress concentration. While detectors of the first group are entirely feasible using modern Josephson junction SQUID devices, the system would be very expensive and very complex to operate. Thus, the project considered only the second type of detector in any detail.

The requirements which were considered in the development of the design are:

1) The device should operate at any temperature from 90 K to ambient and should not be affected in any detrimental way by repeated cycling over this temperature range. If the device parameters are temperature sensitive, a means of determining the temperature should be provided.

2) The portion of the magnetometer which is attached to the tank (magnetometer head) should be relatively insensitive to mechanical shock over the temperature range stated above. The judgment of what represents a sufficient insensitivity is somewhat subjective since most devices respond to the impulse or force-time profile rather than the total force and only limited information on the profiles to be encountered is available.

3) Attachment of the device to the inner tank should be simple; it should operate from one side of the tank, most likely the side facing the insulation space. Because of this also, it should be of reasonable size and weight and require only electrical leads to a plug sealed onto the outer shell of the container.

4) Because of its inaccessible location, the magnetometer head should be capable of in-place calibration, perhaps with an initial calibration before attachment.

5) The magnetometer should be capable of an unambiguous indication of relatively low percentages of martensite. A study of the report by Reed, et al. [1] indicates that quite large amounts of martensite (40-90%) are found before failure occurs when the stress is applied at low temperature - a situation which is the most likely to be the case here. Carrier landing should result in the greatest stress, and this would occur when the tank is full of liquid oxygen.[3] We have chosen a minimum detection level of 3% martensite with an accuracy of ±1% as the design parameter.

6) The magnetometer head should be relatively inexpensive ~$100-200 each, but the operational electronics package, of which only a few would be required, may be somewhat more expensive ~$1000.

7) The operation of the device should be relatively uncomplicated to allow operation by people at the technician level.

3. Systems Studied

As the first part of the project, a brief survey was made of commercially available instruments which might meet the requirements just outlined and none were found. Also, devices which, although not manufacturered commercially, have been used to make magnetic measurements in industry for many years, such as magnetic potentiometers, were considered and discarded. These devices and their principles of operation

---

[3]This assumption needs careful evaluation by the Navy as impact-fatigue failure may occur at $T \geq 250$ K with the production of only very low amounts of martensite, on the order of 1-5%[1]. The detector described here is capable of detection at these levels, but the increased precision required would result in a more sophisticated electronics package than that suggested here.

are described in detail by many authors such as Bozorth[7], Zijlstra[8], and Sanford and Cooper[9].

More modern devices, usually designed for weak field measurement and which are quite sophisticated electronically were also reviewed. These are described in detail in the papers from two recent conferences[10, 11]. Again, for one reason or another, these devices were removed from consideration.

Actual experimental evaluations were made of five classes of devices:

1) Low frequency inductive magnetometer coil in which the change of the self inductance due to the higher permeability of the martensite is detected as a frequency shift.

2) Magnetomorphic effect magnetometer in which the remnant field of the (previously magnetized) martensite is detected by the change in the resistance of a fine wire or film.

3) Magnetoresistive magnetometer which detects the remnant field by exhibiting a change in bulk resistivity at low temperature.

4) A ring-core flux gate magnetometer which detects the imbalance in the magnetizations of two identical ferromagnetic cores caused by the remnant field of the martensite.

5) Hall effect devices which detect the remnant field by measuring the deflection of the charge carrier flow through a semiconductor or semimetal.

All of these devices proved to be unpromising except for the last two, and the flux gate was finally eliminated as being too sensitive to small changes in the ambient magnetic field caused by machinery or large amounts of ferromagnetic material nearby. We feel that with sufficient

study, the flux gate should be a valuable device for martensite detection in another set of circumstances and might well be the most reasonable large area magnetometer.

Our final choice for this application was the class of Hall effect devices. Two magnetometer heads will be described which operate on the same principle but with differing sensitivities and shock characteristics. We feel that these devices represent the best system, given the constraints of the last section.

Magnetic field devices using Hall probe detection are not uncommon. The great majority of these devices were developed some years ago by F. Foerster and are described in detail by McMaster[12]. The actual devices were quite expensive and, most likely, not suitable for use at cryogenic temperatures or under shock conditions.

4. Theory of Operation

The Hall effect device magnetometer head is shown in concept in figure 1.

The bcc martensite in 304 stainless steel is a "hard" ferromagnet, i. e., once it is magnetized by an externally applied field it retains some magnetization when the exciting field is removed. In the magnetometer head, the excitation coil provides a field which is concentrated by the iron cap unit (made from magnetically "soft" iron) into a path which is through the stainless steel region covered by the cap. Near the Hall probe the excitation field is nearly perpendicular to the plane of the probe. When the exciting field is turned off, the Hall probe will detect no remnant magnetic field unless hard ferromagnetic particles were present in the flux path, in this instance martensite.

If magnetized material is present, the exciting coil can be used as a demagnetizing device by application of a steadily decreasing ac

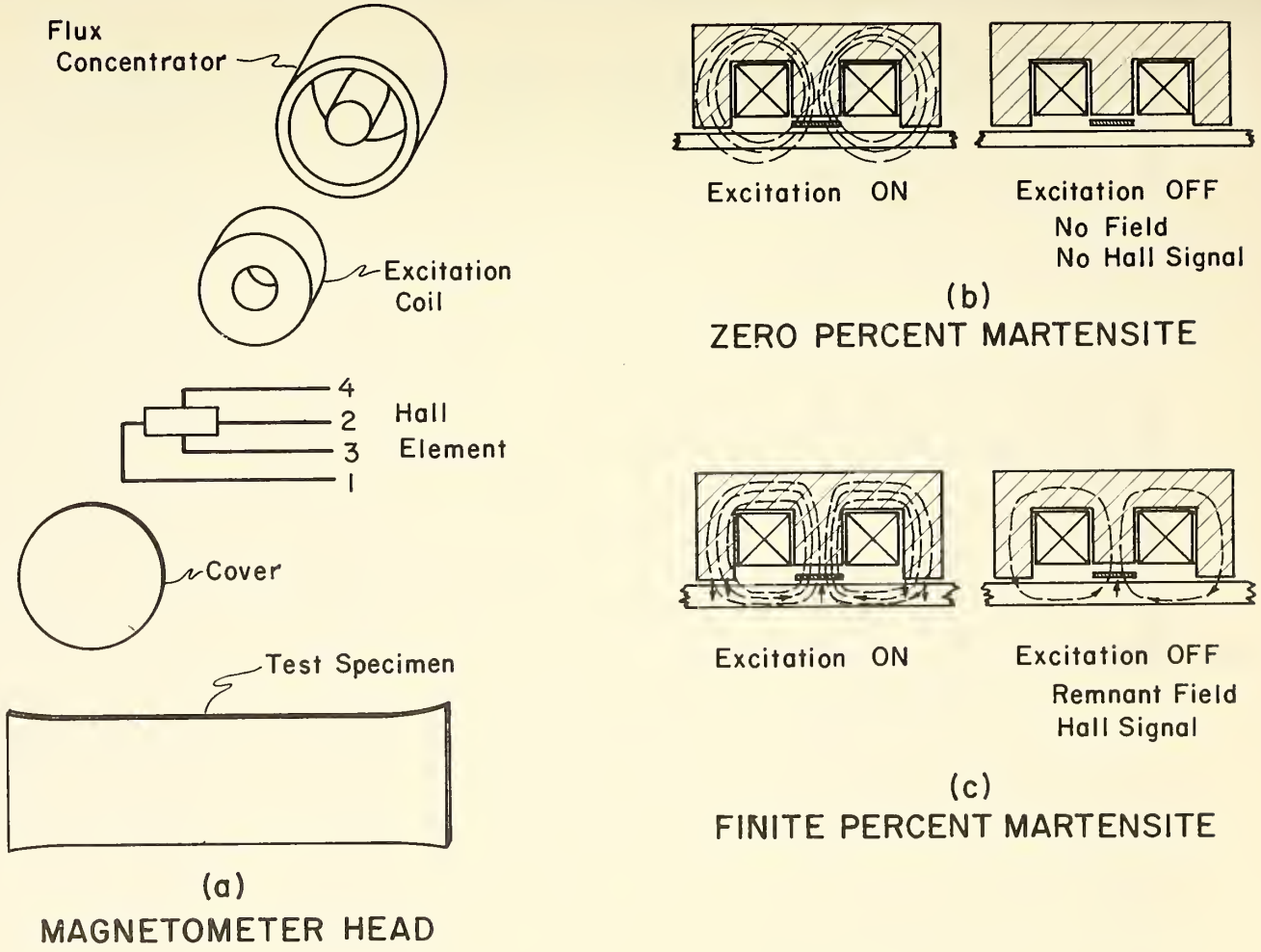

Figure 1. Magnetometer head and magnetization sequence.

voltage to the coil. This step is essential in order to set a zero for the Hall device, since it cannot be easily removed from the tank.

The operation of Hall effect devices is described in detail in many texts[13]; basically, a magnetic field applied perpendicular to an electric current flowing in a strip of metal or semiconductor tends to deflect the carriers (electrons or holes) to the side of the strip, which results in an electric field across the strip perpendicular to both the current and the magnetic field. This is the Hall field; its form for a field normal to the strip is

$$E_H = R_H IB \ (V/m), \tag{1}$$

where I is the current, B the magnetic field and R_H is the Hall constant which in its simplest form is given by

$$R_H = \frac{1}{nec}, \tag{2}$$

where n is the number of carriers, e the electronic charge and c the velocity of light. Obviously a small number of carriers is desirable for large signals and thus semiconductors and semimetals (particularly bismuth) are more common in these devices than pure metals.

5. Design Criteria

There are few absolute criteria required for this device; in fact, its high degree of flexibility is one of the finer features. Here we present a brief commentary on each part of the device and include the data for two actual test devices which were made in order to check out the concepts. The construction of these particular units was based mostly on conveniently available materials and most likely does not represent an optimum for any given application.

The magnetometer consists of two distinct parts, the magnetometer head, shown in figure 1, which attaches to the tank, and the electronics package which is only attached to the head when tests are in progress.

5. 1 The Three Components of the Magnetometer Head

a. The Excitation Coil

This coil is a simple-wound, air core coil and may be of any design whatever. One would like to have the highest field easily obtainable and thus a tightly wound coil is best. For reasonably sized coils, the field at the location of the Hall probe in figure 1 (with the iron flux concentrator removed) will be around H = 100 Oe. The trade off between wire size and excitation current keeps this number nearly constant independent of coil size.

The model units used the coil from a conventional 75 mH rf choke which measured 2. 2 cm o. d. x 1 cm i. d. x 1 cm thick, had a resistance of 160Ω at room temperature and produced a field in air of ~ 100 Oe at an excitation current of 125 mA, which is near the heating limit of the coil.

One should note that the resistance of the pure copper wire from which the coil is wound is decreased by about a factor of 8 from its room temperature value at liquid oxygen temperature and, theoretically, one could increase the current, and thus the field, by this factor. Realistically, an increase of more than a factor of 2-3 should be carefully evaluated as the tight winding of the coil does not allow for rapid heat transfer to the surface and local heating may occur. The coil could be elaborately wound with cooling fins, etc. , but this does not seem to be necessary.

Because the resistance is temperature dependent, the coil may be calibrated and then used with low currents (~ 1-5 mA) as a rough thermometer if and when this is necessary.

b. The Flux Concentrator

This piece is also capable of being constructed in many ways. Its function is to provide a flux path of low magnetic reluctance (analagous to electrical resistance) for the field from the coil at all points exterior to the sheet which is to be measured. The high permeability of the iron from which it is made gives a higher flux density at the Hall element than the excitation coil alone, a rough value being B = 400-500 gauss. The central part of the flux concentrator to which the Hall probe is attached should be larger than the active area of the Hall probe.

This concentrator may be made from any soft iron, i. e., iron which does not retain its magnetization when an applied field is removed. The cylindrical model unit was machined from commercial high purity iron and was 2. 8 cm o. d. x 1. 5 cm high and machined out to take the excitation coil described in (a). In another model the linear yoke structure, of laminated transformer steel, from a 7H choke along with the choke coil was used. It worked well at room temperature, but was very massive and thus took a long time to cool to lower temperature. No delamination of the core was observed on several cycles to 76 K.

In any working model, the pedestal which holds the Hall probe should be recessed with respect to the outer edges of the concentrator so that, when the unit sits on the test plate, the Hall device is not in direct contact with the test plate and thus suffers no stress from this source. However, it should be as close as possible. In the model units, the entire open end of the concentrator was covered with a 0. 007 inch sheet of beryllium copper sealed to the concentrator (brass or any other non-magnetic material will do).

Attachment of the coil to the flux concentrator is accomplished with silicone rubber; it should not be rigidly potted in place without

carefully considering the low temperature behavior of the potting material. Attachment of the Hall device to the pedestal of the flux concentrator is critical as some of the devices tend to curl at low temperatures. Detachment of the device on repeated thermal cycling results in failure of the unit. Any proven low temperature epoxy or insulating varnish will do, but the bond must be thin and the materials properly prepared. The exact bonding agent chosen will depend, of course, on the particular Hall element; frequently, the Hall device manufacturer will recommend the best adhesive for low temperature use.

c. The Hall Device

This is the most critical element of the entire package and, while many elements exist to choose from, one must ascertain from the manufacturer that his unit will meet the requirements outlined below – particularly those relating to mechanical stability under thermal cycling. Our experience has been that, while very expensive elements ($300) are available, much cheaper ones ($10-30) are perfectly adequate for this application. It should also be stressed here that we are not discussing state-of-the-art devices, but those which are commercially available. If the situation required it, Hall devices of much greater sensitivity could be produced.

For this magnetometer, two types of Hall devices were considered – relatively thick film semiconductor and thin film semimetal (bismuth). These probes are available in a wide range of sizes, particularly the bismuth probes which have active areas ranging from 1 inch square to 1/8 inch square. The size chosen for the model was 1/4 inch square for both probes. This size fits nicely on a 3/8 inch diameter pedestal and is easy to attach. Furthermore, the formation process for martensite is such that the 1/4 inch square represents an area which is certain to be representative of the general state of the steel specimen.

The mechanical stability on repeated thermal cycling is good for both devices, but the semiconductor probes tend to be imbedded in a thick epoxy layer which cracks on cycling and destroys the probe. If the manufacturer can and will provide the semiconductor probes with a thin enough coating this problem does not arise.

For probes of the same size, the semiconductor probe has about ten times the sensitivity of the bismuth probe with equal probe currents. Typical values in millivolts output per kilogauss of applied field at room temperature are 25 mV/kOe for semiconductors[4] and 1.5-2.5 mV/kOe for bismuth. For the system as now designed, this means that voltages of 0.2 to 1.0 mV will have to be detected from the semiconductor probe and 0.02 to 0.10 mV from the bismuth probe.

5.2 Temperature Effects

As the temperature is lowered to 76 K, both probes become more sensitive. The sensitivity of the semiconductor probe increases by about 50% and that of the bismuth probe by a factor of 3-5. Repeated thermal shock cycles to 76 K can change the room temperature sensitivity by as much as 20% for the semiconductor and 5% for the bismuth, but the probes always retain a linear output with applied field over the range of interest. This shows that, for accurate measurements, the probe sensitivity must be determined at each test time. Our measurements indicate that the sensitivity change at the lower temperature is nowhere near as sensitive to repeated cycling, being about 5% for the semiconductor probe to 1.5% for the bismuth one. Thus, rough measurements at the lower temperatures could possibly be made without a sensitivity determination.

---

[4] The manufacturer's rated semiconductor probe current (200 mA) caused excessive heating for this application and it was thus halved for all tests, dropping the sensitivity from the rated 50 mV/kOe to 25 mV/kOe.

We should add that our test was definitely a more severe one than would be encountered in actual use and thus the sensitivity changes observed here might be large compared to those found in use.

5.3 Shock Effects

Shock tests at 76 K showed that no significant effect is to be expected on either of the probes. However, as mentioned earlier, since the actual shock profiles to be encountered are not known in any detail, this test must be considered to be qualitative. It is almost certain that, under extreme shock, the bismuth probe would stand up better than the more brittle semiconductor, but we have not reached these shock levels in our tests.

5.4 Electrical

All Hall effect devices, unless manufactured to nearly impossible tolerances, show a voltage indication on the Hall leads when B = 0 due to the fact that the leads do not attach to the strip exactly opposite each other. This is a true resistive voltage which reverses sign when the current is reversed. This voltage may be quite large and is affected by temperature, repeated temperature cycling and shock, but is fortunately easily removed by a simple circuit which will be discussed shortly.

5.5 Electronics

Here a great deal of lattitude is available to the user of the magnetometer depending on the relative amount of ferromagnetic material he wishes to detect and the precision with which he wishes to do it. The essential parts of the electronics package are:

a. Power Supply for the Magnetizing Coil

In our model this is a 125 mA current supply. Stability is not important since the magnetizing current is applied for only about 10

seconds. However, the circuit should be such that no significant current overshoot takes place when the current is first applied to the coil. For calibration purposes and for sensitivity determinations, it is convenient to have the current continuously variable up to the maximum; a three-level supply would also be acceptable.

b. Power Supply for Demagnetizing

This supply should have a current output with an initial value at least that of the magnetizing current and preferably 20% higher and which alternates sign as the current is decreased to zero. The waveform is not important, but frequencies much higher than 60 Hz would have to be looked at in more detail. A good design parameter is a 60 Hz current which decreases uniformly to zero in 30-60 seconds.

c. Power Supply for the Hall Device

Here we require a constant current supply, with about 1% regulation, operating at a single level. If this supply also could operate at a level near 1 mA, it could drive the excitation coil in its function as a thermometer.

d. A Voltmeter for Reading the Output of the Hall Device

The best instrument for this application is a digital millivoltmeter with microvolt precision, an instrument which should be commonly available around electronics installations or which can be purchased for $500-700 for a very stripped down, but perfectly adequate model.

e. A Resistive Zero Circuit for Eliminating the Voltage Offset Inherent in the Hall Devices

One common circuit which works with a wide range of devices is shown in figure 2. Included here is a resistor, R_2, across the Hall voltage leads which is sometimes suggested by Hall probe manufacturers for

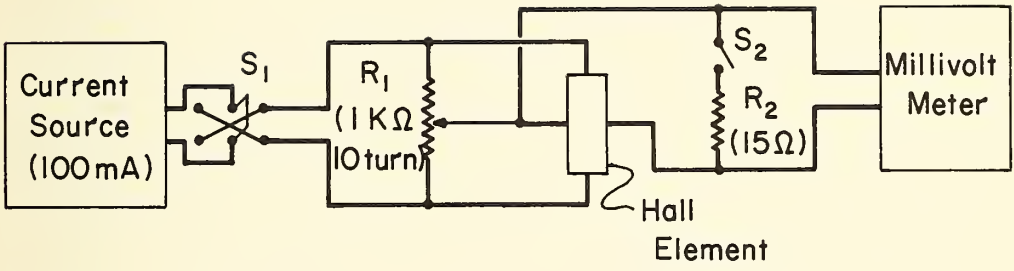

Figure 2. Circuit diagram for resistive voltage offset zero.

optimum linearity of the Hall voltage with field. Also included is a reversing switch, S_1, for the control current which is necessary in order to properly set the resistive zero.

6. Operating Characteristics

Here we present the operating characteristics of the two working models discussed in the last section. These are laboratory tests; any final design should be field tested, particularly for shock.

Since the purpose of the device is detection of various percentages of martensite in austenitic stainless steel, particularly 304, a number of test specimens (11 in all) were prepared by stressing standard tensile blanks at 76 K with a conventional tensile test machine. The percent martensite present in each of these specimens was determined by a gauge which measures the force necessary to pull a calibrated magnet from the surface of the specimen. This device is not particularly accurate, especially at low concentrations of martensite, but it was felt to be sufficient for this purpose. Other methods of determining the percent martensite exist and will be discussed in the last section of this report.

The 304 stainless steel calibration specimens were made from 1/16 inch stock and, when stressed, covered a range of martensite percentage from 1 to 75.

The operation manual for these devices is given below with specific numbers for the model devices. The steps of the operation would be the same for any device built on the principal outlined here, but the numbers, obviously, might be quite different.

Operation Manual

1. Demagnetize with the excitation coil,
   150 mA @ 60 Hz
   decreasing to 0 mA
   in 30-60 s.

2. Set resistive zero of Hall probe,
   by reversal of control current with switch S_1 of figure 2, and adjustment of potentiometer R_1.

3. Magnetize with the excitation coil,
   125 mA dc for 10 s,
   turn off current.

4. Read Hall probe signal.

5. Demagnetize as in step 1.

6. Read Hall probe.
   Should be same reading as step 2.

7. Determine percent martensite from a calibration curve.
   These curves are determined before the magnetometer head is mounted by measurements on test specimens, the production of which will be discussed shortly. [5]

If temperature measurement is desired or necessary:

Before step 1, apply a 1 mA current to the excitation coil and read the voltage. Read T from a calibration chart.

---

[5] If an in-place calibration is anticipated, an extended series of measurements using these specimens is required before mounting. This procedure is outlined in the appendix.

If determination of probe sensitivity is necessary, it may be done by the relatively complex sequence of measurements which are outlined in the appendix. This complexity arises because the device cannot be removed from the tank. It is well worth determining if recalibration is actually going to be necessary under the thermal cycling to be found in practice; there is an excellent chance that such will not be the case. It would also be worthwhile to consider the possibility of mounting one device on an area of the tank which is unlikely to undergo transformation and use it as a calibration comparator. Another possible way to avoid recalibration is to recognize that an indication of, say 5 ±2% martensite, is quite adequate as a go-no go determination since much higher percentages will actually occur before failure is likely. The probes tested never varied more than that in sensitivity, even on 140 cycles of direct immersion into liquid nitrogen (76 K) from room temperature – a test condition much more stringent in terms of thermal shock than would be encountered in use.

Curves from the model devices are shown in figures 3 through 6. Figures 3 and 5 show typical sensitivity curves for the magnetometer heads alone in terms of exitation current. Figures 4 and 6 show the martensite detection capabilities of the two devices.

The curves of figures 4 and 6 are, of course, for a particular device, for a particular excitation current and for a particular sensitivity. Changing any of these parameters would require adjustment of the vertical scale. In particular, a sensitivity increase of the probe from S_1 to S_2 requires that the observed Hall voltage be multiplied by the factor (S_1/S_2) before the corresponding percent martensite is read from the curve.

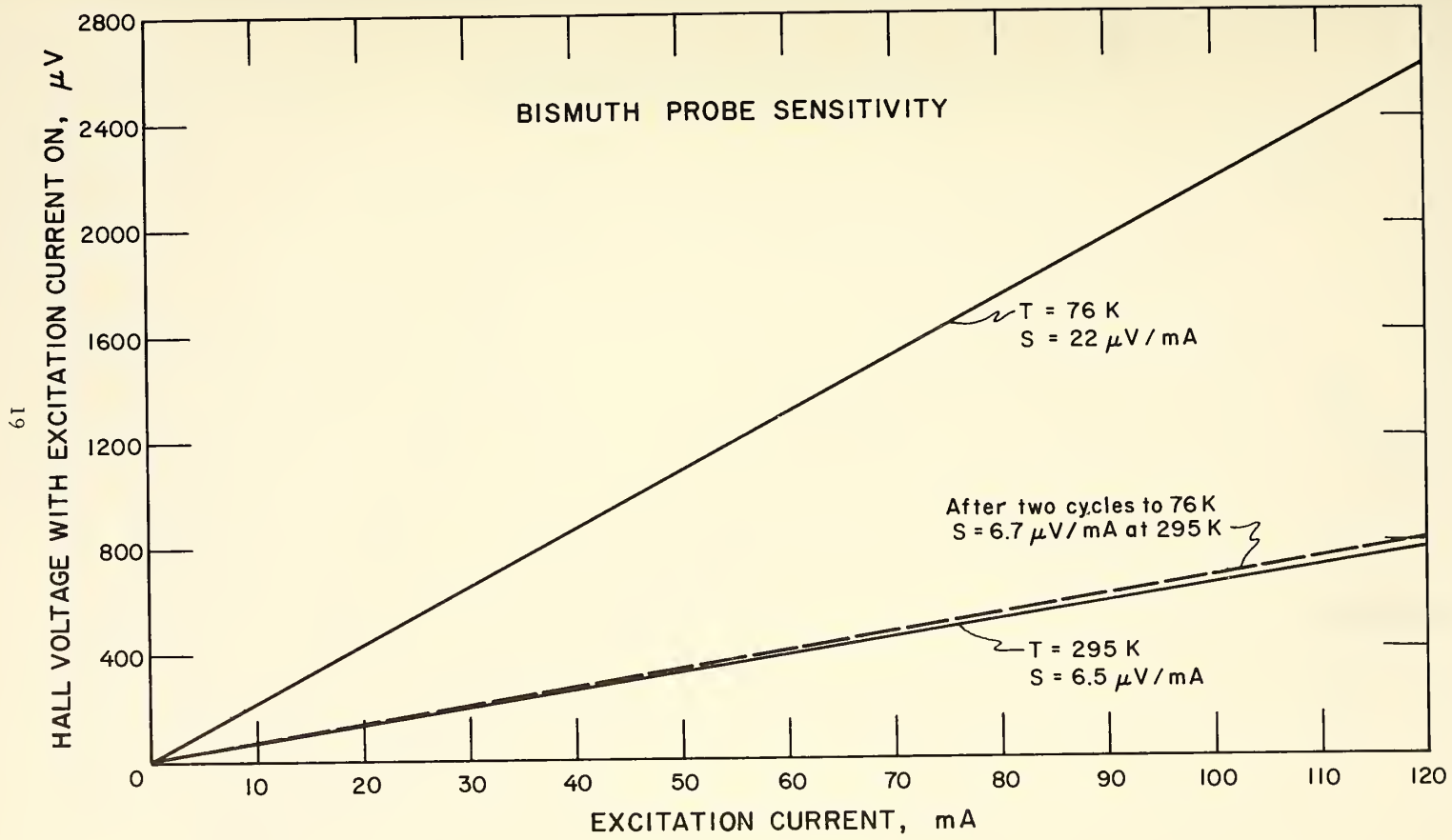

Figure 3. Sensitivity of bismuth probe (typical).

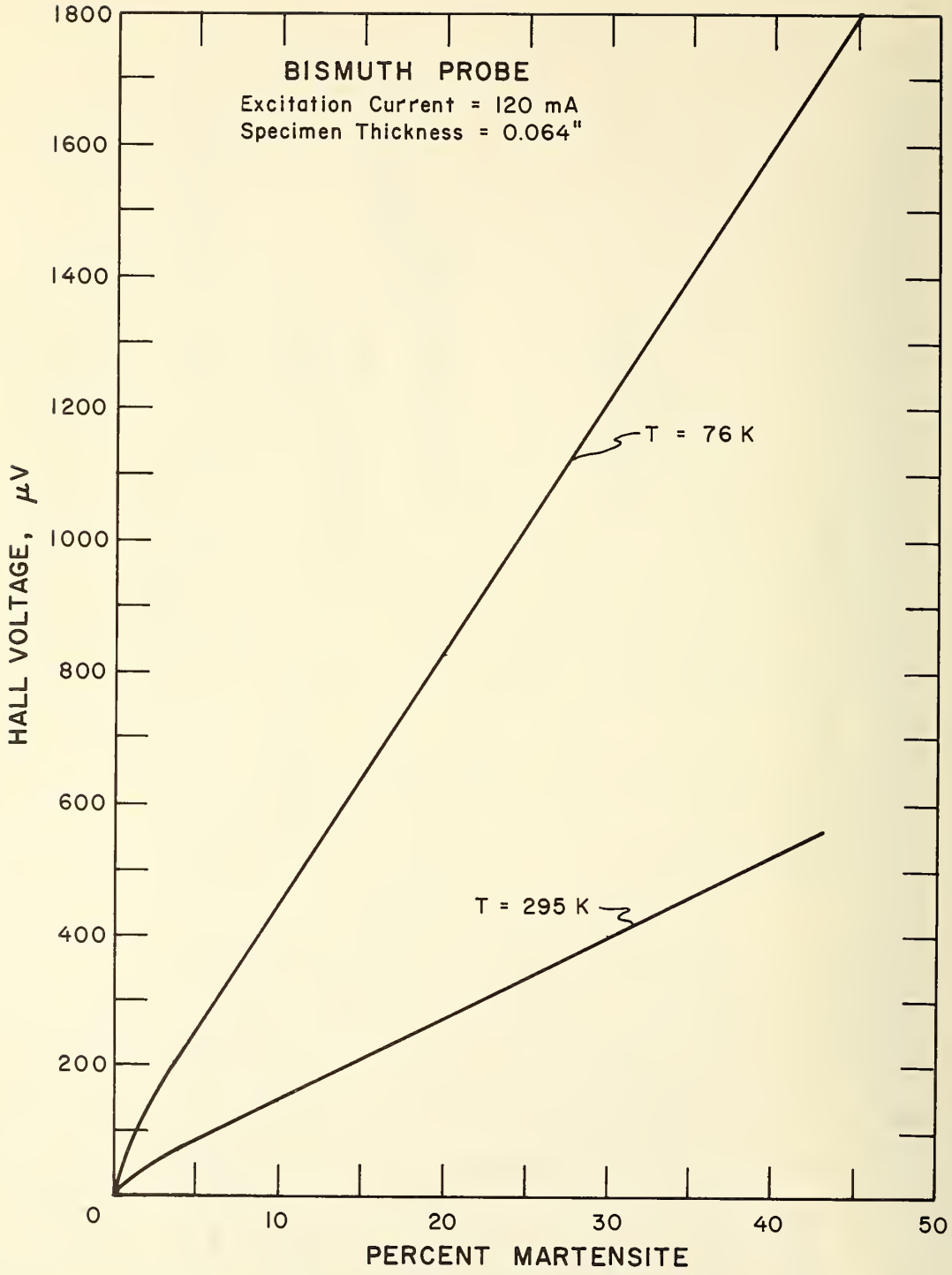

Figure 4. Detection of percent martensite by bismuth probe (typical).

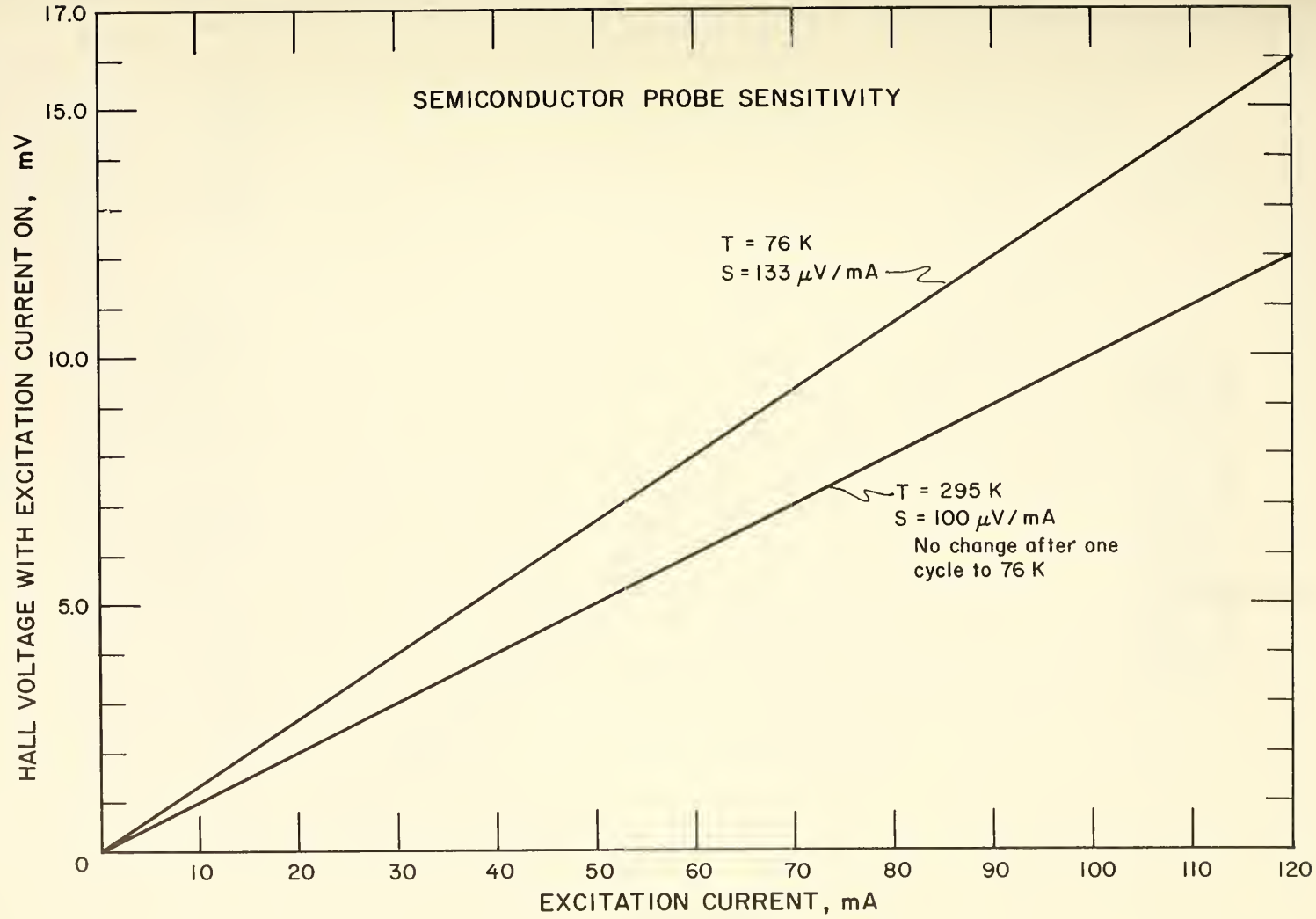

Figure 5. Sensitivity of semiconductor probe (typical).

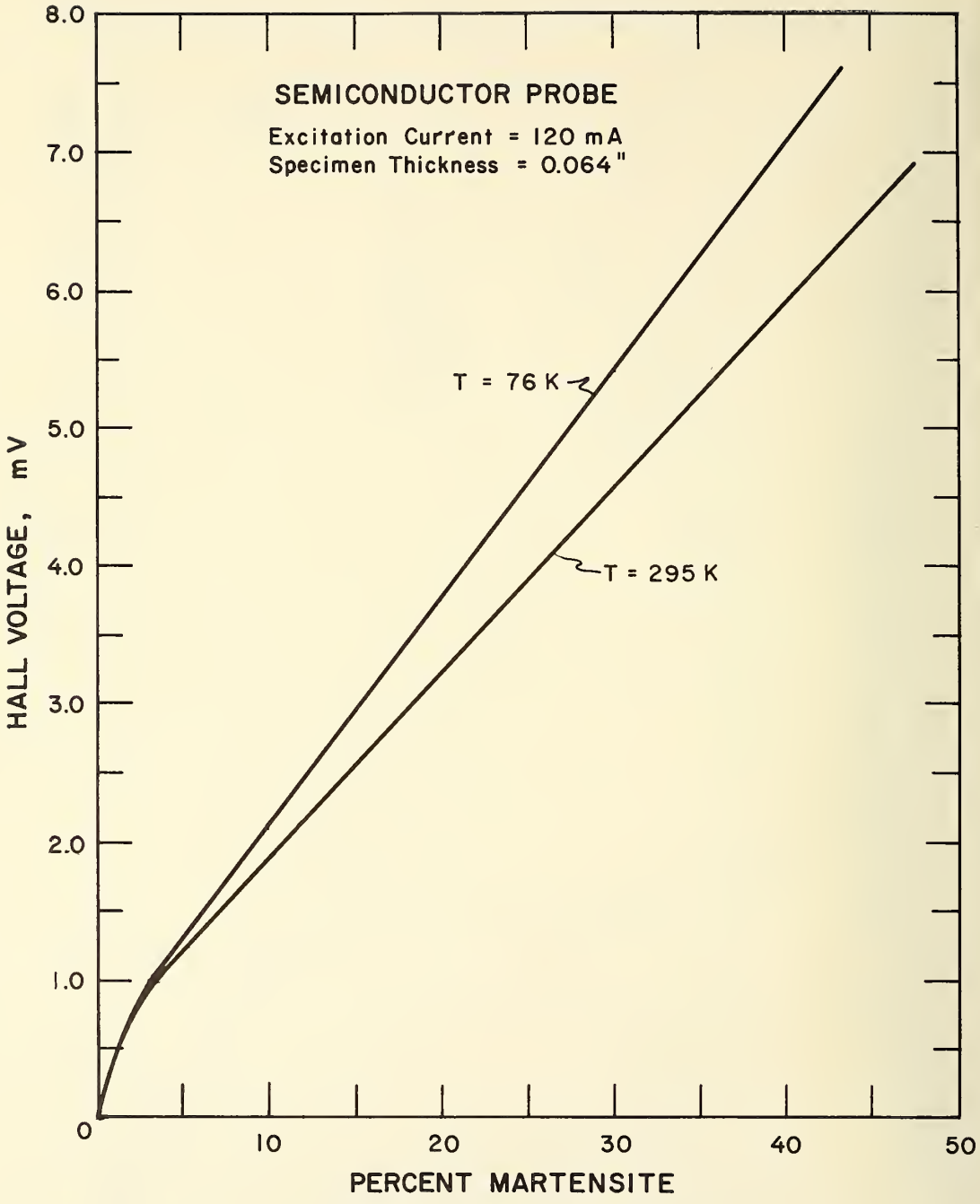

Figure 6. Detection of percent martensite by semiconductor probe (typical).

The response of this detector, as of all others, is sensitive to the thickness of the plate on which it is placed, i. e., a 3/16 inch thick plate with approximately one percent martensite gives a reading nearly two times that of a 1/16 inch plate. This means that each detector must be calibrated, at least at room temperature, with calibration specimens of the same thickness as the plate on which it is to be used. There is an alternative to this – a calibration of reading vs thickness vs percent martensite, but this is by no means a linear function.

7. Further Development

In this section we present the steps which are necessary to transform these design criteria into a field device. Some of these have already been discussed, particularly as regards the electronics package and will not be repeated here in any detail.

We feel that there are two programs which need to be carried out before immediate application is made of this device. The first is development of dependable test specimens of stainless steel of the same thickness as the tank wall. These specimens should be prepared in the same manner as ours but, in addition, should have the percentage of martensite determined with greater accuracy. This can be accomplished either by x-ray techniques[2] or optical microscopy[3] or perhaps, by comparison with test specimens now used in the steel industry. The second consideration is simply a test of the device under conditions of use or conditions closely approximating the same. Ideally, one would like to attach several magnetometer heads to a tank so that they could be removed, and then subject the whole thing to a realistic test simulating stresses perhaps a factor of two or three greater than the maximum to be expected in service. Liquid nitrogen could be used as the fluid for such a test. This test would also

serve to indicate exactly where on the tank the points of high stress are located, as the tank could be mapped by a magnetometer in as much detail as necessary.

In addition to these, calibration curves vs temperature for the particular Hall element and excitation coil used in the final design will be necessary if measurements are to be made at temperatures other than ambient and 90 K. Also, our assumption as to the temperature at which the maximum shock loading is going to take place needs to be investigated.

Not discussed here, but uppermost in our minds after this program, is the possibility of further development and sophistication of this basic device as an all-purpose, non-destructive testing system. Our research, and conversations with experts in the field, have convinced us that this basic concept could have wide applicability to a number of very difficult test problems.

8. References

1. R. P. Reed, R. L. Durcholz, R. E. Schramm, and T. J. Patrician, Nat. Bur. Stands., Tech. Note 609 (1971).
2. R. P. Reed and C. J. Guntner, Trans. Met. Soc. AIME 230, 1713 (1964).
3. R. P. Reed, Acta Met. 10, 865 (1962).
4. R. P. Reed and R. P. Mikesell in: Advances in Cryogenic Engineering, Vol. 4, K. D. Timmerhaus, Ed., Plenum Press, New York (1960), p. 84.
5. C. J. Guntner and R. P. Reed, Trans. ASM 55, 399 (1962).
6. F. Schwartzberg, "Cryogenic Materials Data Handbook", MLTDR-64-280 (PB171809), Clearinghouse for Tech. and Sci. Inform., Washington, D. C. (1968).
7. R. M. Bozorth, Ferromagnetism, D. vanNostrand, N. Y. (1951).
8. H. Zijlstra, Experimental Methods in Magnetism 1. Generation and Computation of Magnetic Fields, Interscience, N. Y. (1967).
9. R. L. Sanford and I. L. Copper, National Bureau of Standards Monograph 47 (1962).
10. Rev. Phys. Appl. 5, 1 (1970).
11. J. Appl. Phys. 42, 27 (1971).
12. R. C. McMaster, Nondestructive Testing Handbook, Vol. II., Ronald Press, N. Y. (1959). Sections 33 and 34.
13. C. Kittel, Introduction to Solid State Physics, Third Edition, John Wiley and Sons, N. Y. (1967), p. 242.

Appendix

Here we describe a method by which changes in the calibration of a magnetometer head Hall probe may be determined when the probe is in place. If the magnetometer head is cycled a few times between the temperature extremes to be encountered in use before its initial calibration, significant changes in calibration are not likely. It is strongly recommended that the cycling be done, as the calibration procedure is not extremely accurate unless great care is taken in preparation of the initial curves.

Before the magnetometer head is mounted, a graph similar to figure 7 should be made in the laboratory using carefully prepared test specimens. Specimens should be available for every 5% martensite over the entire range and every 1-2% below 10%. Note that this graph is identical to the calibration curve of figure 6 except that the reading of the Hall probe with the excitation current on is also plotted. It is not expected that these curves will be perfectly linear when made using test specimens of accurately known martensite concentration. A separate graph is required for each temperature at which the probe is to be used and for each excitation current.

Assume now that the probe, with initial sensitivity S_1 represented by V_H = 10.6 mV @ 0% martensite with the 120 mA excitation on, has been mounted on the tank for some time. Some martensite may be present in the tank wall.

To determine if the calibration has changed:

a) Measure V_H with excitation on and measure V_H with excitation off.

b) Plot these values on the graph.

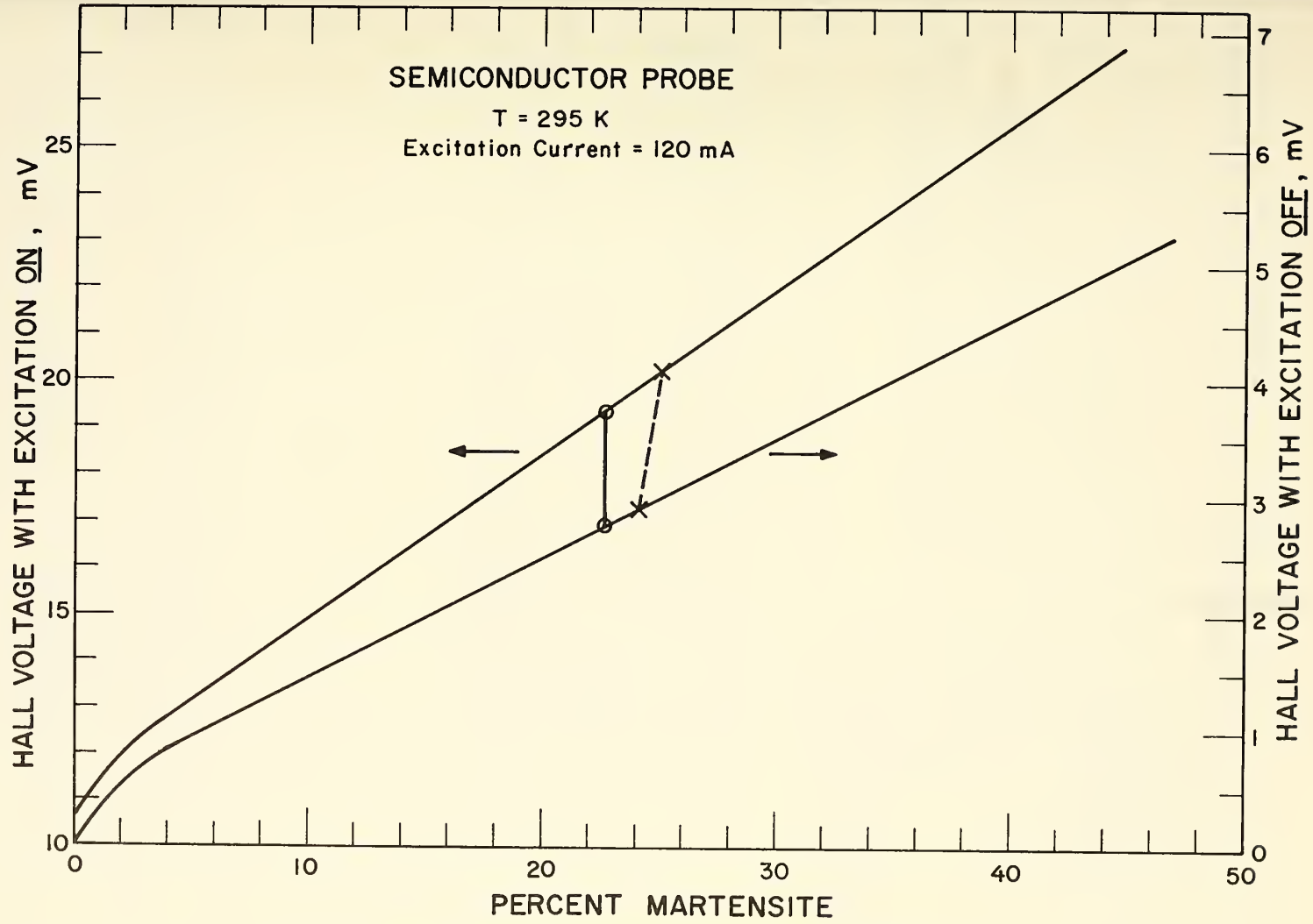

Figure 7. In-place probe calibration technique.

As an example, we show the results of such a measurement by the points labeled X.

If the points do not fall nearly on a vertical line, the calibration has changed and the probe calibration can be determined as follows:

a) Determine what factor each of the V_H readings must be multiplied by to make the points fall on a vertical line. This is most easily done by trial and error. In our example the factor is 0. 955 and the resulting V_H values are shown by the circles.

b) This factor is the ratio of the old sensitivity to the new sensitivity and all readings from the probe should be multiplied by this factor before being compared to the calibration curve to determine the percent of martensite. In our example the new sensitivity would be

$$\frac{10.6}{0.955} = 11.1 \text{ mV @ 0 percent martensite,}$$

and the specimen has ~23% martensite vs 24% which would be the reading with no recalibration.

FORM NBS-114A (1-71)

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET	1. PUBLICATION OR REPORT NO. NBS-TN-613	2. Gov't Accession No.	3. Recipient's Accession No.

4. TITLE AND SUBTITLE

Martensite Transformation Detection in Cryogenic Steels (Magnetometer Development)

5. Publication Date: December 1971

6. Performing Organization Code

7. AUTHOR(S)

F. R. Fickett

8. Performing Organization

9. PERFORMING ORGANIZATION NAME AND ADDRESS

NATIONAL BUREAU OF STANDARDS
DEPARTMENT OF COMMERCE
Boulder, Colorado 80302

10. Project/Task/Work Unit No.

11. Contract/Grant No.
Project Order
1-8022

12. Sponsoring Organization Name and Address

Naval Air Engineering Center
Philadelphia, Pennsylvania

13. Type of Report & Period Covered
N.A.

14. Sponsoring Agency Code

15. SUPPLEMENTARY NOTES

16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)

This report presents design criteria for a magnetometer device developed to determine the martensite concentration in 304 stainless steel. Specifically, the device is designed to monitor the cold tank of an airborne liquid oxygen container which is subject to periodic stress pulses. The amount of martensite is relatable to the plastic strain and thus provides a mechanism for the prediction of impending failure.

The magnetometer, developed after a critical review of the constraints, is an excitation coil-Hall effect system. The system is very versatile in design and is easily adapted to particular problems. It is relatively insensitive to thermal and mechanical shock and easily capable of detecting martensite at the 1% level.

17. KEY WORDS (Alphabetical order, separated by semicolons)

Fatigue monitor; low temperature; magnetometer; martensite; nondestructive.

18. AVAILABILITY STATEMENT

[X] UNLIMITED.

[ ] FOR OFFICIAL DISTRIBUTION. DO NOT RELEASE TO NTIS.

19. SECURITY CLASS (THIS REPORT)	21. NO. OF PAGES
UNCLASSIFIED	31
20. SECURITY CLASS (THIS PAGE)	22. Price
UNCLASSIFIED	40

NBS TECHNICAL PUBLICATIONS

PERIODICALS

JOURNAL OF RESEARCH reports National Bureau of Standards research and development in physics, mathematics, chemistry, and engineering. Comprehensive scientific papers give complete details of the work, including laboratory data, experimental procedures, and theoretical and mathematical analyses. Illustrated with photographs, drawings, and charts.

Published in three sections, available separately:

• Physics and Chemistry

Papers of interest primarily to scientists working in these fields. This section covers a broad range of physical and chemical research, with major emphasis on standards of physical measurement, fundamental constants, and properties of matter. Issued six times a year. Annual subscription: Domestic, $9.50; $2.25 additional for foreign mailing.

• Mathematical Sciences

Studies and compilations designed mainly for the mathematician and theoretical physicist. Topics in mathematical statistics, theory of experiment design, numerical analysis, theoretical physics and chemistry, logical design and programming of computers and computer systems. Short numerical tables. Issued quarterly. Annual subscription: Domestic, $5.00; $1.25 additional for foreign mailing.

• Engineering and Instrumentation

Reporting results of interest chiefly to the engineer and the applied scientist. This section includes many of the new developments in instrumentation resulting from the Bureau's work in physical measurement, data processing, and development of test methods. It will also cover some of the work in acoustics, applied mechanics, building research, and cryogenic engineering. Issued quarterly. Annual subscription: Domestic, $5.00; $1.25 additional for foreign mailing.

TECHNICAL NEWS BULLETIN

The best single source of information concerning the Bureau's research, developmental, cooperative, and publication activities, this monthly publication is designed for the industry-oriented individual whose daily work involves intimate contact with science and technology—*for engineers, chemists, physicists, research managers, product-development managers, and company executives.* Annual subscription: Domestic, $3.00; $1.00 additional for foreign mailing.

NONPERIODICALS

Applied Mathematics Series. Mathematical tables, manuals, and studies.

Building Science Series. Research results, test methods, and performance criteria of building materials, components, systems, and structures.

Handbooks. Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

Special Publications. Proceedings of NBS conferences, bibliographies, annual reports, wall charts, pamphlets, etc.

Monographs. Major contributions to the technical literature on various subjects related to the Bureau's scientific and technical activities.

National Standard Reference Data Series. NSRDS provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated.

Product Standards. Provide requirements for sizes, types, quality, and methods for testing various industrial products. These standards are developed cooperatively with interested Government and industry groups and provide the basis for common understanding of product characteristics for both buyers and sellers. Their use is voluntary.

Technical Notes. This series consists of communications and reports (covering both other agency and NBS-sponsored work) of limited or transitory interest.

Federal Information Processing Standards Publications. This series is the official publication within the Federal Government for information on standards adopted and promulgated under the Public Law 89–306, and Bureau of the Budget Circular A–86 entitled, Standardization of Data Elements and Codes in Data Systems.

Consumer Information Series. Practical information, based on NBS research and experience, covering areas of interest to the consumer. Easily understandable language and illustrations provide useful background knowledge for shopping in today's technological marketplace.

NBS Special Publication 305, Supplement 1, Publications of the NBS, 1968-1969. When ordering, include Catalog No. C13.10:305. Price $4.50; $1.25 additional for foreign mailing.

Order NBS publications from:

Superintendent of Documents
Government Printing Office
Washington, D.C. 20402

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
Washington, D.C. 20234

OFFICIAL BUSINESS

Penalty for Private Use, $300

POSTAGE AND FEES PAID
U.S. DEPARTMENT OF COMMERCE

U.S.MAIL